10 PRIMER RESIDUE ANALYSIS

10.1 Analytical Approach

Generate one or more Primer Residue Evidence Handling Worksheets (Appendix 19). For results data set purposes, each Primer Residue kit is to be considered a separate item. Open the Primer Residue kit and label each of the sample vials. Visually examine stub surface. If debris is present go to Section 10.2, Carbon Evaporation or use the ASPEX VP 2000 in VP mode. Otherwise, proceed to Section 10.3, Automated Scanning Electron Microscope/Energy Dispersive X-ray System (SEM/EDS) analysis.

10.1.1 Minimum Standards and Controls

10.1.1.1 Primer Residue kits are ordered by DFS to our specifications. When a lot of Primer Residue kits arrives, two percent of the kits must pass QC inspection before any kits from that lot are released to User Agencies.

10.1.1.2 A visual inspection is made of the Primer Residue kits to note whether all components of the kit are present. Any visible debris on the collection surface is noted. One sample from each kit is analyzed via automated SEM/EDS. A copy of the Primer Residue SEM/EDS Worksheet and Analysis Summary Sheet is retained. Samples from these kits become future negative control samples for automated Primer Residue runs.

10.2 Carbon Evaporation

10.2.1 Purpose

10.2.1.1 Carbon evaporation, or coating, makes the sample electrically conductive and reduces charging in the SEM. In addition, carbon does not interfere with EDS analysis.

10.2.1.2 Special Considerations that should be noted with this technique: Carbon evaporation reduces charging on samples containing visible debris. Therefore, the overall automated SEM/EDS run time may be shortened due to reduction in charging of the sample.

10.2.2 Safety Considerations

10.2.2.1 Carbon evaporation produces a bright arc at the carbon rod tip source. During evaporation, the carbon rod should only be viewed through an appropriate welder's glass.

10.2.3 Minimum Standards and Controls

10.2.3.1 A negative control is included with all Primer Residue samples to be coated for a given run. If a single sample requires carbon coating then all samples in that particular Primer Residue automated run will be coated.

10.2.3.2 A glass slide is used to evaluate the coating.

10.2.4 Analytical Procedures

10.2.4.1 Label the bottom of the sample stubs before coating samples.

10.2.4.2 Operate the carbon evaporator following the manufacturer's operations manual.

10.2.4.3 Slowly rotate the sample plenary stage during the coating procedure.

10.2.5 References

10.2.5.1 Gabriel, B. L. SEM: A User's Manual for Materials Science. 1985, pp.156 -161. ISBN: 0-87170-202-9**.**

10.3 Automated SEM/EDS Primer Residue Analysis, ASPEX: PSEM75/2000, VP2000, 3000

10.3.1 Purpose

10.3.1.1 Automated SEM/EDS is used to search samples for potential primer residue particles. These instruments search a user defined area at a magnification of 500X on each sample. Generally speaking, a particle highly specific to primer residue is defined as a particle with a spherical or molten appearance (noncrystalline) containing the elements: Pb, Ba and Sb. Particles indicative of primer residue would have similar morphology and include 2 of the 3 elements listed above. The VP2000 instrument can be operated in either high vacuum mode for electrically conductive samples or in variable pressure mode at 0.2 Torr for nonconductive samples.

10.3.1.2 This is a sensitive, non-destructive technique used to detect the primer residue component of gunshot residue.

10.3.2 Safety Considerations

10.3.2.1 The EDS detector system must be cooled with liquid nitrogen. Insulated gloves and safety glasses shall be worn when filling any Dewar.

10.3.2.2 During filament replacement be sure to disable the beam power supply located on the back panel of the beam supply under the bias knob.

10.3.2.3 Be aware of elevated temperatures when changing a filament which has been in operation.

10.3.2.4 If operating in variable pressure mode, change to high vacuum mode before changing the filament. In addition, always turn the beam off before changing vacuum modes.

10.3.3 Minimum Standards and Controls

10.3.3.1 The EDS detector is electronically calibrated prior to an automated primer residue run. The Cu Lα peak should be symmetrical through the peak centroid line as should the Cu Kα peak. The centroids of these peaks will be monitored during the automated run. As a matter of QA these peaks should be within 0.03 KeV of the nominal values. FWHM of Cu Kα will also be monitored during the automated run. It should not exceed 170 eV. If any of these values are exceeded recalibrate the EDS detector.

10.3.3.2 Before an automated primer residue run can begin, the SEM filament must be saturated at 20 KV and aligned with the manual gun centering knobs on a copper standard where beam damage will not occur. Also, electronically center the filament by monitoring the BSED brightness and contrast of the copper standard in the line scan mode. Adjust the filament centering in the advanced filament application.

10.3.3.3 A positive control consists of a sampling device containing known primer residue particles.

10.3.3.4 The backscatter detector threshold is set to detect primer residue particles ≥ 1 micron in diameter.

10.3.3.5 A QC analysis is run on the same area on the positive control at the beginning of the run, after 3 samples have been examined and at the end of the run. After the run is complete 10 particle images and their corresponding spectra from each of the positive controls is generated showing primer residue particles found in the 1.0 micron size range. These sheets are stored in a QC notebook in the SEM lab.

10.3.3.6 In every automated SEM/EDS primer residue run, a negative control is analyzed just before the final positive control field. The negative control is the first sample to be loaded in the SEM and the last sample to be removed after the run is complete. Negative control samples can be reused providing they have not been carbon coated. On or about the 1st of each month the current negative control sample will be discarded. A carbon coated negative control sample will be discarded after the automated primer residue run due to loss of adhesive properties during the carbon coating procedure.

10.3.3.7 Approximately once a month the photographic unit of the SEM is checked using an ASTM measurement standard and a gold standard on carbon. Apertures are replaced if the image cannot be stigmated.

10.3.4 Analytical Procedures

10.3.4.1 A primer residue analysis setup is performed by setting up or using an existing .afa file.

10.3.4.2 A new automated run is setup. DHD6.ZRR and GSRCHEM139.VCF files are selected. QC analysis is run in Analysis 1 position and typically after every 3 case samples as well as after the negative control at the end of the run. Once the setup has been created it is printed out and saved.

10.3.4.3 Load samples into the SEM as described in primer residue analysis setup sheet.

10.3.4.4 The SEM is pumped down to operational vacuum.

10.3.4.5 The filament is saturated at 20 KV and allowed to stabilize for at least 15 minutes.

10.3.4.6 The beam is placed on a copper standard and the spot size adjusted to achieve approximately 1,500 CPS with a process time of 25.6 µsec. A spectrum from 0 to 10.24 KeV is collected and calibrated using the EDS detector calibration software.

10.3.4.7 The backscatter electron detector is set to detect particles ≥ 1.0 micron in diameter in the following manner: The stage is driven to the QA field on the primer residue standard sample. An area is located where 1.0 micron particles are visible at 1000X in the right hand image. This image is focused and stigmated using the mini imaging and medium scan speed mode. Imaging is then switched to normal and high scan speed.

10.3.4.8 Magnification is dropped to 100X and the sample moved to an area where copper and carbon are present and a threshold image is acquired. Particles in the 1.0 micron size range must be in the green detection area. This is achieved by keeping the brightness at -0% (average noise at 16 in linescan mode) and adjusting the contrast accordingly.

10.3.4.9 Each sample to be run is then checked using a three point focus system through the stage setup program. It is imperative that the sample coordinate positions are defined in exactly the same order as listed on the primer residue setup sheet. Areas of the stub where the aluminum surface is exposed should be avoided.

10.3.4.10 The magnification is returned to 100X. The area containing the 1.0 micron particles on the primer residue standard is focused as described in step 7. The quality check is reviewed and set to check copper before the first, after the last and between every 1 sample.

10.3.4.11 The automated run is started. Data is stored in the next sequential run file.

10.3.4.12 The primer residue standard field is monitored to ensure 1.0 micron particles are being detected. If they are not, the backscatter image and/or focus must be readjusted.

10.3.4.13 Each sample surface is searched until 10 potential particles highly specific to primer residue are found or until the entire predesignated area is searched or until three hours of search time has elapsed.

10.3.4.14 When the automated run is finished (typically the following day) a calibration is done on copper to check the EDS detector as described earlier.

10.3.4.15 The SEM/EDS parameters are set up as listed on the "Primer Residue Worksheet SEM/EDS".

10.3.4.16 The operator performs a manual confirmation of the potential primer residue particles detected during the automated run.

10.3.5 References

10.3.5.1 Andrasko, J. and Maehly, A. C., "Detection of Gunshot Residue by Use of the Scanning Electron Microscope," Journal of Forensic Sciences, Vol. 22, 1977, pp. 279-287.

10.3.5.2 ASTM E 1588 – 95 (Reapproved 2001) Standard Guide for Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive Spectroscopy.

10.3.5.3 Basu, S., "Formation of Gunshot Residues," Journal of Forensic Sciences, Vol. 27, 1982, pp. 72-91.

10.3.5.4 DeGaetano, D. H., Siegel, J. A., and Klomparens, K. L., "A Comparison of Three Techniques Developed for Sampling and Analysis of Gunshot Residue by Scanning Electron Microscopy and Energy Dispersive X-Ray Analysis," Journal of Forensic Sciences, Vol. 37, 1992, pp. 281-300.

10.3.5.5 Garofano, L., Capra, M., Ferrari, F., Bizzaro, G. P., DiTullio, D., Dell'Olio, M., and Ghitti, A., "Gunshot Residue Further Studies on Particles of Environmental and Occupational Origin," Forensic Science International, Vol 103, 1999, pp. 1 – 21.

10.3.5.6 Mosher, P. V., McVicar M. J., Randall E. D., and Sild, E. H. "Gunshot Residue-Similar Particles Produced by Fireworks", Canadian Society of Forensic Science Journal, Vol 31 (2) 1998, pp. 157-168.

10.3.5.7 Nesbitt, R. S., Wessel, J. E., and Jones, P. F., "Detection of Gunshot Residue by Use of the Scanning Electron Microscope," Journal of Forensic Sciences, Vol. 21, 1976, pp. 595-610.

10.3.5.8 Sild, E. H. and Pausak, S., "Forensic Applications of SEM/EDX," Scanning Electron Microscopy, Vol. 2, 1979.

10.3.5.9 Wolten, G.M., Nesbitt, R.S., Calloway, A.R., Loper, G.L. and Jones, P.F., "Final Report on Particle Analysis for Gunshot Residue Detection," Report ATR-77(7915)-3. The Aerospace Corp., Sept. 1977.

10.3.5.10 Wright, D., and Trimpe, M., "Summary of the FBI Laboratory's Gunshot Residue Symposium May 31-June 3, 2005", Forensic Science Communications, Vol. 8, (3) 2006.

10.3.5.11 Gunshot Residue V2.00 from 1.12 Documentation, Operator's Manual. R. J. Lee Instruments Limited. 515 Pleasant Valley Rd., Trafford, PA 15085.

10.4 Documentation

10.4.1 Elements detected in potential primer residue particles and the diameter of the particles are documented on the output.

10.4.2 The original Primer SEM/EDS Worksheet will be retained in the case file associated with the 1st case sample in the automated run.

10.4.3 A representative photograph and corresponding EDS spectrum for each confirmed primer residue particle is generated for the case file. EDS spectra should be obtained at 1000 counts full scale or 99 seconds Live Time whenever possible.

10.4.4 Spectral files and image files (where applicable) from the representative particles are saved to the hard drive and downloaded for archiving purposes.

10.4.5 The Primer Residue Evidence Handling Worksheet is used to provide documentation for the results reported in the Certificate of Analysis. "Boiler plate" wording on the Worksheet that is not applicable may be struck through with a single line and the entire paragraph bracketed and initialed by the examiner.

10.4.6 The QA output must include a positive control field before and after the casefile sample/s. A copy of the negative control must also be included in the casefile or QA binder (where applicable).

10.4.7 An Analysis Summary Report for each of the case samples analyzed is also included in the casefile.

10.4.8 The PRIMER RESIDUE ANALYSIS INFORMATION FORM that is provided in the Primer Residue kit is retained by the laboratory and kept in the casefile.

10.4.9 The outside of the Primer Residue kit is photocopied and retained in the casefile.

10.4.10 In suicide cases, either the right or left hand sample is analyzed first (based upon information on the RFLE or PRIMER RESIDUE ANALYSIS INFORMATION FORM). If more than one primer residue particle is found on the sample, the second sampling device is not analyzed.

10.4.11 If the PRIMER RESIDUE ANALYSIS INFORMATION FORM indicates that the amount of time between the shooting event and the sampling for a living individual is in excess of 8 hours the samples will not be analyzed unless extenuating circumstances exist.

10.5 Report Wording

To the maximum extent possible, report wording will be selected from the following:

10.5.1 No primer residue particles were found on the samples in Item _____ marked right hand or left hand.

The absence of primer residue on the hands is consistent with an individual not having fired a weapon. A negative result could also occur from circumstances such as: washing the hands, wiping the hands, wearing gloves, sweating profusely, environmental factors including wind and rain, bloody hands, excessive debris on the sample, greater than 4 to 6 hours passing between firing and sampling, or the weapon not producing primer residue on the hands when discharged.

10.5.2 Particles highly specific to primer residue were found on the samples in Item _____ marked right/left hand.

Primer residue can be deposited on the hands by circumstances such as: firing a weapon, handling a weapon, being in the proximity to the discharge of a weapon or coming into contact with an object that has primer residue on it. The examination itself cannot determine the relative likelihood of these listed circumstances.

10.5.3 Particles indicative of primer residue were found on the samples in Item _____ marked right/left hand. Indicative particles are less specific to, but commonly found in, primer residue.

Primer residue can be deposited on the hands by circumstances such as: firing a weapon, handling a weapon, being in the proximity to the discharge of a weapon or coming into contact with an object that has primer residue on it. The examination itself cannot determine the relative likelihood of these listed circumstances.

10.5.4 It was indicated on the Primer Residue Analysis Information Form that the amount of time between the shooting event and the sampling for Item _____ was approximately _____ hours and _____minutes. Item _____ was not analyzed.

It should be noted that the optimal window of opportunity for sampling the hands of a living individual lasts for up to 4 to 6 hours after the shooting event.

10.5.5 The sample in Item _____ marked right/left hand was not necessary for analysis.

♦End